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(71) Applicant(s)
Robert McBride Ltd
(Incorporated in the United Kingdom)
Middleton Way, Middleton, MANCHESTER, M24 4DP,
United Kingdom

(72) Inventor(s)
Robert Eric Williams
Mark Phillip Houghton

(74) Agent and/or Address for Service
Robert McBride Ltd
Park Road (West Side), BARROW-IN-FURNESS,
Cumbria, LA14 4BN, United Kingdom

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US 5531910 A **US 4614831 A**

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(54) Abstract Title
Deodorising cleaner

(57) A malodour counteracting system comprises a cyclohexane derivative in combination with a hydrophilic polymer. Such a combination gives surprisingly efficacious cleaning results as measured by malodour reduction. Even more effective malodour counteractancy is obtained by the inclusion of surfactants, particularly anionic and amphoteric surfactants, and cyclodextrins where further beneficial effects are observed.

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Deodorising cleaner

The present invention concerns the use of deodorising chemicals for use in household products. In particular household cleaning products where a combination of deodorising technologies in combination with detergent additives has shown surprisingly efficacious results.

Air fresheners are an established part of the consumer products market and are formulated with a variety of fragrances. One particular reason for their use is to mask undesirable odours and this is achieved by a number of mechanisms. Masking malodour using large amounts of any fragrance is possible as the adsorption of malodour molecules on to the nasal membrane will be reduced by competing fragrance molecules and the overall sensory stimulation will be higher masking the malodour. From everyday experience it is evident that such over use of fragrance is undesirable and may simply relace one undesirable odour by another.

Work in recent years has therefore focussed on alternative routes to malodour suppression. The use of perfume accords, when one fragrance molecule works co-operatively with another in sensory terms to give a combined odour, different from the constituents, is one route. Molecules which form a positive accord with malodour molecules are utilised, the sensory perception of malodours being converted by virtue of the accord in to a positive odour. This is known technology and is routinely used by the skilled perfumer. Whilst the materials added for this purpose may have an inherently pleasant smell this is not necessary as disclosed in US 4, 719, 105, US 4, 622, 221 (BBA) where various particularly effective cyclohexanol derivatives are disclosed. This disclosure makes no reference to the combination of such materials in household cleaners and in particular no reference is made to their use with surfactants or polymers. In particular no indication of any co-operative or synergistic benefit of combining such materials is disclosed. This approach to malodour counteractancy suffers from the disadvantage that the problem is simply masked and since aroma perception differs between people will only provide a partial solution.

An alternative approach is to incorporate substances capable of adsorbing or absorbing malodour components, activated carbon is well known for this but being black is not suitable for use in cleaning compositions. Cyclodextrins may also act as odour adsorbers. Their use in uncomplexed

form has been disclosed in cleaning compositions (WO 96/05358, Procter) where nonionic surfactants are described as an optional ancillary aid to help solubilisation. No disclosure of anionic surfactants or of polymers is made. In particular no indication of any co-operative or synergistic benefit of combining such materials is disclosed. This approach to malodour counteractancy suffers from the disadvantage that the odour is only neutralised when it diffuses to the non-volatile adsorbent. Its effect is therefore limited.

Surprisingly we have found that a combination of high molecular weight non-volatile polymers and/or surfactants in combination with cyclohexyl alcohol derivatives and even more so when combined with cyclodextrins gives particularly efficacious malodour removal properties in cleaning compositions.

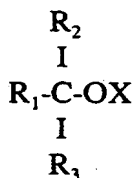
A detergent composition with malodour counteractancy is claimed, comprising,

- a) 0.1 to 10% of a hydrophilic polymer of molecular weight greater than 1,000
- b) 0.00001 to 1% of a volatile cyclohexane derivative.
- c) 0 to 25% of a surfactant.
- d) 0 to 5% of a Cyclodextrin.

The detergent composition may utilise any of the known forms, solid (as powder, tablet, granulate), liquid (as aqueous, non-aqueous, atomised spray) or intermediate forms such as a gel, paste or wax.

The preferred form of the invention is as a dilute liquid, preferably one in which the polymer imparts thixotropic properties. The liquid should have a viscosity of between 0.5 and 500 mPa.s, more preferably between 1 and 100mPa.s at a shear rate of 100s^{-1} and preferably a viscosity higher than this at a shear rate of 1s^{-1} , most preferably an increase in viscosity of more than 25% most preferably of more than 100% is achieved at the lower shear rate.

Cyclohexane derivatives are claimed of the formula



Where R₁ is an unsubstituted cyclohexane ring, X is H or an alkyl or ester (R₄CO-). R₂ and R₃ are independently selected from H, OH, OR₄, or an C₁ to C₄ alkyl. R₄ is a hydrocarbon moiety of MW between 29 and 1000. Wherein the boiling point of the liquid is below 500°C at ambient pressure. Further examples of suitable materials are described in US 4, 719, 105 (BBA). These materials are included at levels of between 0.00001% to 1% of a product, more preferably between 0.0001 to 0.1%, even more preferably from 0.0005 to 0.01% by weight of the total detergent product. Use of neat perfumes in aerosols is an exception to this where a level of 10 times higher for each range is suitable as the propellant is not usually described as being part of the composition.

The cyclohexane derivative will typically be formulated as part of a perfume composition. Such compositions are formulated by the trade for their aesthetic qualities, their chemical stability, cost and the presence of novel perfume accords by the perfumer skilled in the art. It is considered within the scope of the invention to include other such perfumery components as may be deemed suitable. It is considered beneficial to include in such compositions for malodour counteraction so called pro perfumes which are typically high molecular weight low volatile species which degrade in a defined way, usually activated by a consumer or machine activity, to yield a low molecular weight fragrance.

It is considered obvious that someone skilled in the art can use a cyclohexane malodour counteracting derivative as part of a pro fragrance. In particular one with a labile ester or amide link which can be broken by the enzymes present in the bacteria that give rise to malodour by decomposing other molecules in refuse, sweat etc. to release the odour counteracting species.

The perfume ingredients and compositions of this invention include the conventional ones

known in the art. Selection of any perfume component, or amount of perfume, is based on aesthetic considerations. Suitable perfume compounds and compositions can be found in the art including U.S. Pat. Nos. 4,145,184, Brain and Cummins, issued Mar. 20, 1979; 4,209,417, Whyte, issued Jun. 24, 1980; 4,515,705, Moeddel, issued May 7, 1985; and 4,152,272, Young, issued May 1, 1979, all of said patents being incorporated herein by reference. Many of the art recognized perfume compositions are relatively substantive, as described hereinafter, to maximise their odour effect on substrates. However, the use of Cyclodextrin/perfume complexes may be used to improve substantivity and longevity of perfume. A substantive perfume is one that contains a sufficient percentage of substantive perfume materials so that when the perfume is used at normal levels in products, it deposits a desired odour on the treated fabric. In general, the degree of substantivity of a perfume is roughly proportional to the percentage of substantive perfume material used. Relatively substantive perfumes contain at least about 1%, preferably at least about 10%, substantive perfume materials. Substantive perfume materials are those odorous compounds that deposit on substrates via the treatment process and are detectable by people with normal olfactory acuity. Such materials typically have vapour pressures lower than that of the average perfume material. Also, they typically have molecular weights of about 200 or above, and are detectable at levels below those of the average perfume material. Perfumes can also be classified according to their volatility, as mentioned hereinbefore. The highly volatile, low boiling, perfume ingredients typically have boiling points of about 250 .degree. C. or lower. Many of the more moderately volatile perfume ingredients are also quickly lost. For example, substantially all of such perfumes are lost in the drying cycle of a typical laundry process. The moderately volatile perfume ingredients are those having boiling points of from about 250 .degree. C. to about 300 .degree. C. The less volatile, high boiling, perfume ingredients referred to hereinbefore are those having boiling points of about 300.degree. C. or higher. A significant portion of even these high boiling perfume ingredients, considered to be highly substantive, can be lost, during a laundry drying cycle, and it is desirable to have means to retain more of these ingredients on the fabrics. Many of the perfume ingredients as discussed hereinafter, along with their odour characters, and their physical and chemical properties, such as boiling point and molecular weight, are given in "Perfume and Flavour Chemicals (Aroma Chemicals)," Steffen Arctander, published by the author, 1969, incorporated herein by reference. Examples of the highly volatile, low boiling, perfume ingredients are: anethole, benzaldehyde, benzyl acetate,

benzyl alcohol, benzyl formate, iso-bornyl acetate, camphene, cis-citral (neral), citronellal, citronellol, citronellyl acetate, para-cymene, decanal, dihydrolinalool, dihydromyrcenol, dimethyl phenyl carbinol, eucalyptol, geranial, geraniol, geranyl acetate, geranyl nitrile, cis-3-hexenyl acetate, hydroxycitronellal, d-limonene, linalool, linalool oxide, linalyl acetate, linalyl propionate, methyl anthranilate, alpha-methyl ionone, methyl nonyl acetaldehyde, methyl phenyl carbinyl acetate, laevo-menthyl acetate, menthone, iso-menthone, myrcene, myrcenyl acetate, myrcenol, nerol, neryl acetate, nonyl acetate, phenyl ethyl alcohol, alpha-pinene, beta-pinene, gamma-terpinene, alpha-terpineol, beta-terpineol, terpinyl acetate, and vertenex (para-tertiary-butyl cyclohexyl acetate). Some natural oils also contain large percentages of highly volatile perfume ingredients. For example, lavandin contains as major components: linalool; linalyl acetate; geraniol; and citronellol. Lemon oil and orange terpenes both contain about 95% of d-limonene. Examples of moderately volatile perfume ingredients are: amyl cinnamic aldehyde, iso-amyl salicylate, beta-caryophyllene, cedrene, cinnamic alcohol, coumarin, dimethyl benzyl carbinyl acetate, ethyl vanillin, eugenol, iso-eugenol, flor acetate, heliotropine, 3-cis-hexenyl salicylate, hexyl salicylate, lilial (para-tertiarybutyl-alpha-methyl hydrocinnamic aldehyde), gamma-methyl ionone, nerolidol, patchouli alcohol, phenyl hexanol, beta-selinene, trichloromethyl phenyl carbinyl acetate, triethyl citrate, vanillin, and veratraldehyde. Cedarwood terpenes are composed mainly of alpha-cedrene, beta-cedrene, and other C.sub.15 H.sub.24 sesquiterpenes. Examples of the less volatile, high boiling, perfume ingredients are: benzophenone, benzyl salicylate, ethylene brassylate, galaxolide (1,3,4,6,7,8-hexahydro- 4,6,6,7,8,8- hexamethyl- cyclo- penta- gama-2-benzopyran), hexyl cinnamic aldehyde, lyral (4-(4-hydroxy-4-methyl pentyl) -3-cyclohexene-10-carboxaldehyde), methyl cedrylone, methyl dihydro jasmonate, methyl-beta-naphthyl ketone, musk indanone, musk ketone, musk tibetene, and phenylethyl phenyl acetate.

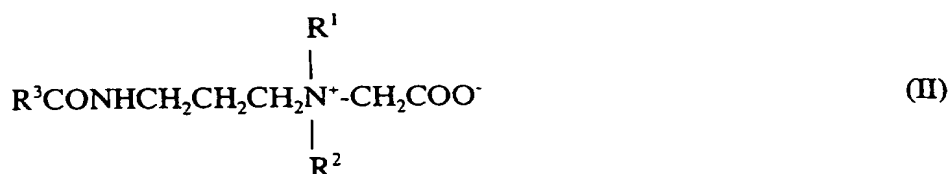
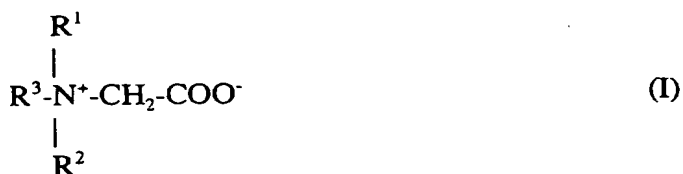
Polymeric additives are claimed which are substantially hydrophilic such that they can be dissolved at more than 10g/l in water at 25°C. A solution of 1% of the polymer in water at 25°C should have a viscosity of between 1 and 10000 mPa.s, more preferably between 1 and 500mPa.s even more preferably between 1 and 100mPa.s at a shear rate of 100s⁻¹ and preferably a viscosity higher than this at a shear rate of 1s⁻¹, most preferably an increase in viscosity of more than 25% most preferably of more than 100%.

Suitable polymeric materials known in the art are Hydrogenated castor oils (eg Croduret 50 ex Croda), acrylic acid polymers (eg ex National Starch) Acrylate maleate polymers (eg Sokolan CP-5 ex BASF) and poly ethylene glycols (eg ex Hoechst), poly vinyl pyrrolidone (eg K50 ex ISP), Carbopol (ex 3V). Suitable levels of polymer inclusion are between 0.1 to 10% more preferably between 0.2 and 2% most preferably between 0.3 and 1.2%.

The detergent formulation in accordance with the invention will include at least one surface active agent which may, for example, be an anionic, cationic, non-anionic or amphoteric surface active agent. Any of the surface active agents widely used in detergent formulations may be employed in the present invention.

If an amphoteric surface active agent is used it may be present in the formulation in an amount of 0.1 to 10% by weight, more preferably 0.5 to 5%, even more preferably 1 to 4% on the same basis.

The amphoteric surface active agent may be betaine surface active agent. Preferred betaines may be either of the formula (I) or (II).



In the above formula, R^1 and R^2 may be the same or different C_{1-4} alkyl groups whereas R^3 is an alkyl group having 8-22 carbon atoms, more preferably 12 to 18 carbon atoms e.g. mixed C_{10} to C_{14} .

The preferred betaine for use is cocoamidopropyl betaine.

An alternative amphoteric surface active agent for use in the formulation of the invention is a glycinate of the formula



where R^3 is as defined above.

Other suitable materials are as given in chapter 1 of "Amphoteric Surfactants", e.g. Lomax Ed, Marcel Decker, New York 1996.

It is highly preferred that a cationic surface active agent is employed in conjunction with the amphoteric surface active agent. The cationic surface active agent is preferably used in an amount of up to 2% by weight of the formulation and is conveniently added in conjunction with the clay. Examples of suitable cationic surface active agents include quaternary ammonium salts having three lower (C_{1-4}) alkyl groups (preferably methyl groups) and a long chain (C_{8-20}) alkyl group, e.g. coco trimethyl ammonium chloride. Further examples include alkyl pyridinium salts and other compounds in which the nitrogen atom of the pyridine assumes a quaternary form, e.g. as in an alkyl pyridinium bromide.

Further examples of cationic surface active agents which may be used include amine and imidazoline salts.

If an anionic surface active agent is used then it is preferably present in the formulation in an amount of up to 20%, more preferably up to 10%, even more preferably up to 5% by weight of the formulation. Examples of anionic surface active agents which may be employed include alkylaryl sulphonates, alkyl sulphates, ether sulphates and ether carboxylates all as conventionally employed in laundry detergent formulations.

If a non-ionic surface active agent is used then it is preferably present in an amount of up to 20%

by weight of the formulation, more preferably 2 to 10% on the same basis. Examples of non-ionic surface active agent which may be used include alkoxylates, ethylene oxide/propylene oxide block copolymers, alkanolamides (e.g. monoethanolamides and diethanolamides), esters and amine oxides.

The surfactant may be omitted or more preferably can be present at up to 25%, more preferably between 1% and 20%. For textile washing compositions the most preferable range is between 10 and 20% and for hard surface cleaning between 1 and 5% and for fabric refresher and air freshener products less than 1%. The preferred surfactant is one carrying an electrical charge, most preferably an amphoteric or anionic surfactant.

Suitable Cyclodextrin materials are described in WO 96/05358, and are incorporated herein by reference. Due to the complexity of surfactant based systems the person skilled in the art would experiment with various commercial Cyclodextrin to find which performed the best. Cyclodextrins are an optional but preferred embodiment of the invention. Cyclodextrins, Cyclodextrinoids, optionally as alcohol, amide, ether, ester, hydrophobised, conjugated, granulated, encapsulated and solubilised, derivitised and embodied as such are all envisaged as ways of changing their use provided that they retain some ability to complex smaller molecules. Cyclodextrin incorporation of between 0 and 5%, more preferably between 0.05 and 2%, most preferably between 0.075 and 1.25% is within the scope of the invention.

The formulation may include at least one builder salt in a total amount of 0.1% to 50% by weight of the formulation. The builder may be for example be an alkali metal phosphate or alkali metal carbonate. A particularly preferred builder is sodium triphosphate. Other suitable builders are zeolites, citric acid or sodium citrate. It is recognised that zeolites may have an auxiliary, even predominant function as co-adsorbers of odours though their effect is considered as being simply additive.

A bleaching composition may also be included. The preferred bleaching system for use in the invention comprises a hydrogen peroxide precursor compound and the bleach activator as known in the art which is capable of reacting with the hydrogen peroxide to generate a peracid. The

hydrogen peroxide precursor compound may, for example, be an inorganic persalt e.g. a perborate (in the monohydrate and/or tetra hydrate form), a percarbonate or a persulphate. The alkali metal salts of these compounds are preferred, particularly sodium and potassium salts. Alternatively in the case where the detergent formulation is in solid form, the bleaching agent may be a urea-hydrogen peroxide complex. In the case of a liquid formulation the hydrogen peroxide precursor compound may be hydrogen peroxide per se. Pre-formed per acids known in the art are also considered suitable.

A clay may be used in the composition, either *per se* or as a carrier for the perfume. The clay which is used in the formulation of the invention may be any one of the fabric softening clays having fabric softening properties used in laundry detergent formulations. Such clays are generally of the "lamellar type" and are such that the layers "separate" to become deposited on the garments being washed. The clay may for example be a Smectite such as a Laponite, Bentonite, Montmorillonite, Hectorite or Saponite. For example, the clay may be a Sodium Montmorillonite, a Sodium Hectorite, a Sodium Saponite, a Calcium Montmorillonite or a Lithium Hectorite.

An enzyme may be included in the composition. The enzyme may, for example, be a protease, amylase, lipase or cellulase (or mixtures thereof) such as commonly used in detergent formulations. Examples of suitable enzymes are available under the names Opticlean, Savinase, Esperase; Termamyl, Maxamyl, Lipomax, Lipolase; Celluzyme and Carezyme. The amount of enzyme incorporated in the formulation will depend on activity but will typically be 0.1 to 3%. This level is particularly suitable for Savinase 6.0T, Termamyl 60T, Celluzyme 0.7T and Lipomax.

It will be appreciated that the formulation may incorporate additional components as conventionally included in a hard surface cleaner, laundry detergent, fabric refresher, fabric conditioner or similar product.

A liquid composition is preferably delivered as a spray or aerosol derived from a pressurised aerosol canister or from a hand actuated spray, preferably of the trigger spray variety as sold by,

for instance, Guala UK Ltd.

The invention is illustrated by the following non-limiting examples.

Formulations are prepared by adding the components to water, stirring and equilibrating for 18 hours in a closed container before carrying out any further tests. The Cyclodextrin when used with perfume was pre-mixed with the perfume so as to complex some perfume materials.

Component	A1	A2	A3	A4	A5	A6
Croduret 50 Special (ex Croda)	0.50	1.0	1.0	1.0	1.0	1.0
Citric acid (ex Aldrich)	0.12	0.5	0.5	0.5	0.5	0.5
Sodium triphosphate (ex A&W)	0.15	0.6	0.6	0.6	0.6	0.6
Formalin (40% solution) (ex Aldrich)	0.20	0.2	0.2	0.2	0.2	0.2
Perfume BBA LU 65327 (ex BBA containing Cyclohexane derivative	0.10					
Sodium alkyl benzene sulphonate (ex Huls)		3	3	3	3	0
Perfume White Line (ex Pheonix fragrances UK)		0.1		0.1		
White line with 5% Cyclohexyl ethyl propionate added			0.1		0.1	0.1
Cyclodextrin hydrate (CA No. 10016-20-3 ex Aldrich)				0.1	0.1	0.1
Water	to 100%					

Comparative formulations

Component	C1	C2	C3	C4	C5	C6
Croduret 50 Special (ex Croda)	1.0					
Citric acid (ex Aldrich)	0.5	0.5	0.5	0.5	0.5	0.5
Sodium triphosphate (ex A&W)	0.6	0.6	0.6	0.6	0.6	0.6
Formalin (40% solution) (ex Aldrich)	0.2	0.2	0.2	0.2	0.2	0.2
Perfume BBA LU 65327 (ex BBA containing Cyclohexane derivative						
Sodium alkyl benzene sulphonate (ex Huls)	3		3			
Perfume White Line (ex Pheonix fragrances UK)				0.1		0.1
White line with 5% Cyclohexyl ethyl propionate added		0.1	0.1			
Cyclodextrin hydrate (CA No. 10016-20-3 ex Aldrich)					0.1	0.1
Water	to 100%					

Malodour counteractancy testing

Preparation of odiferous surfaces

Fabric - Pieces of thin white cotton fabric measuring 10cm by 10cm are hung in a household waste storage 'Room' (2m square, 2m high) for 1 week with assorted household waste in open topped plastic bags of about 1M³ capacity, 3 of containing about 30kg of waste at 20°C. The cloths are placed in a sealed polythene bag before removal and used within 24 hours.

'Worktop' - 10cm by 10cm sections of plastic covered kitchen worktop (ex B&Q) were abraded lightly with 00 grade emery paper until the surface shine was lost. The edges of the cut materia are sealed with wax to stop water adsorption. A mixture of 75% Lard and 25% olive oil was made by heating and stirring at 80°C. To this is added 0.01% propanoic acid and 0.001%, 1,4 diamino butane (in a fume cupboard!). The mix was then cooled and 0.2g spread over a circle of 4cm radius of the 'worktop'.

Cleaning procedure

The prepared odiferous substrate are hung vertically and sprayed with the prepared composition using a Guala had held trigger spray with a pump volume per spray of approximately 0.5ml. The surface is sprayed three times and then left to stand for one minute. The surface is then wiped clean/dry with two large paper towels (worktop) or rinsed in 50ml of water (fabric) and then left to air dry for 10 minutes. The odour of the surface is then recorded according the scheme below by five panellists, an average score was taken. To facilitate comparisons the samples are prepared sequentially in a well ventilated area and then moved to another ventilated area for drying and sequential evaluation by personnel not in contact with the cleaning process (ie desensitised).

Scoring system

No malodour	mild malodour	distinct malodour	disagreeable malodour	unavoidable malodour	Barely tolerable
0	1	2	3	4	5

Best -----> Worst

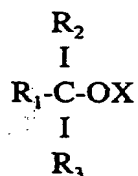
Component	A1	A2	A3	A4	A5	A6
Residual Surface Odour	0.4	2.8	1.2	0.4	2.4	0.6
Residual Fabric Odour	0.8	3.0	1.6	0.6	2.4	0.8

Component	C1	C2	C3	C4	C5	C6
Residual Surface Odour	4	2.0	1.8	3.6	3.2	3.2
Residual Fabric Odour	4.2	2.4	2.0	3.8	3.2	3.8

Result C3 with Synperonic A3 (ex ICI) nonionic surfactant in place of alkyl benzene sulphonate surfactant, score, 1.8, with coco imido propyl betaine (ex Akzo) amphoteric surfactant in place score 1.2.

Claims

- 1) A detergent composition with malodour counteractancy is claimed, comprising,
 - a) 0.1 to 10% of a hydrophilic polymer of molecular weight greater than 1,000
 - b) 0.00001 to 1% of a volatile cyclohexane derivative.
 - c) 0 to 25% of a surfactant.
 - d) 0 to 5% of a Cyclodextrin.
- 2) A composition according to claim 1 wherein the volatile cyclohexane has the formula



Where R1 is an unsubstituted cyclohexane ring, X is H or an alkyl or ester (R₄CO-). R2 and R3 are independently selected from H, OH, OR₄, or an C₁ to C₄ alkyl. R₄ is a hydrocarbon moiety of MW between 29 and 1000. Wherein the boiling point of the liquid is below 500°C at ambient pressure.

- 3) a composition according to claim 1 or 2 comprising containing from 1 to 20% of a surfactant.
- 4) a composition according to claim 3 containing from 2 to 10% surfactant.
- 5) a composition according to claim 3 or 4 wherein the surfactant is an anionic or amphoteric surfactant.
- 6) a composition according to claim any of claims 1 to 5 containing from 0.05 to 5% of a Cyclodextrin or derivative.
- 7) a composition according to claim any of claims 2 to 5 containing from 0.05 to 1% of a Cyclodextrin or derivative.

8) a composition according to claim 2 comprising

- a) 0.3 to 1.2% of a hydrophilic polymer of molecular weight greater than 1,000.
- b) 0.0005 to 0.01% of a volatile cyclohexane derivative.
- c) 1 to 5% of an anionic surfactant.
- d) 0.75 to 1.25% of a Cyclodextrin.

9) a composition according to claim 8 used for the cleaning of solid or fibre surface for the removal of malodorous spoils when applied using a spray or aerosol wherein the composition also contains from 0.1 to 50% of a builder salt.

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Application No: GB 9919353.4
Claims searched: 1 - 9

Examiner: Michael Conlon
Date of search: 24 November 1999

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.Q): C5D (D123)

Int Cl (Ed.6): C11B, C11D

Other: Online: EPODOC, WPI, PAJ

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	US5531910 (Procter & Gamble) Example 9	1
A	US4614831 (International Flavors) Example XIX	1

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

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